

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
15 November 2001 (15.11.2001)

PCT

(10) International Publication Number
WO 01/85482 A1

- (51) International Patent Classification⁷: **B60J 10/04**,
B32B 27/08, 27/32, E05D 3/02 [US/US]; 1465 Gulf of Mexico Drive, #B105, Longboat
Key, FL 43228 (US).
- (21) International Application Number: PCT/US01/14621 (74) Agent: **GOLRICK, Mary, E.**; Calfee, Halter & Gris-
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OH 44114 (US).
- (22) International Filing Date: 7 May 2001 (07.05.2001)
- (25) Filing Language: English (81) Designated States (*national*): CA, CN, CZ, JP, KR, US.
- (26) Publication Language: English (84) Designated States (*regional*): European patent (AT, BE,
CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC,
NL, PT, SE, TR).
- (30) Priority Data:
09/567,911 10 May 2000 (10.05.2000) US
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CORP, INC.** [US/US]; P.O. Box 537012, Sacramento, CA
95853-7012 (US). Published:
— with international search report
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- For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes and Abbreviations" appearing at the begin-
ning of each regular issue of the PCT Gazette.*



WO 01/85482 A1

(54) Title: METHODS OF COATING RUBBER WITH SOLVENTLESS CRYSTALLINE POLYOLEFIN COATINGS

(57) Abstract: A novel laminate useful for seals particularly for vehicle window seals that are flexible, wear resistant and have a low coefficient of friction, is provided. The laminate comprises a rubber substrate and a polyolefin coating disposed on and adherent to the rubber substrate. The invention also provides novel methods of making laminates, particularly seals. Such method comprises the following steps: providing a rubber substrate; then applying a powdered crystalline polyolefin to the rubber substrate, in an amount sufficient to form a continuous layer when melted; and then melting the powdered crystalline polyolefin to form a continuous polyolefin coating disposed on and adherent to the rubber substrate. The invention also relates to seals comprising: a polyolefin rubber body; and a continuous coating of fused polyolefin disposed on and adherent to the rubber seal. Preferably the continuous coating of fused polyolefin is disposed in the glass run channel in the rubber body.

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METHODS OF COATING RUBBER WITH SOLVENTLESS CRYSTALLINE POLYOLEFIN
COATINGS

Background of the Invention

5 Many vehicle seals are flexible to conform to vehicle glass to seal out the elements. The flexibility must be maintained over a wide range of temperatures. Moreover, the seal area in contact with glass requires a low coefficient of friction so that when glass is raised or lowered, the seal does not stick to the glass. While
10 ethylene-propylene-diene monomer rubber (EPDM) has a suitable flexibility, it has a less than preferred coefficient of friction. In an attempt to reduce the coefficient of friction, the surface of ethylene-propylene-diene monomer rubber has been coated with polyurethane. However, the polyurethane coating is not particularly
15 resistant to wear, which results in failure of the seal within a relatively short time. Attempts have been made to produce seals with polymeric coatings other than polyurethane; however such seals typically employ volatile organic solvents during the manufacturing process. Such volatile organic solvents have recently become the
20 subject of governmental regulation.

It would be desirable to have a flexible, wear resistant seal, having a low coefficient of friction, and which is applied without volatile organic solvents.

Summary of the Invention

25 The present invention provides novel laminate useful for seals, particularly for vehicle window seals that are both flexible and wear resistant and have a low coefficient of friction, preferably with an initial coefficient of friction below 0.5. The laminate comprises a rubber substrate and a polyolefin coating disposed on and adherent to
30 the rubber substrate.

The invention also provides novel methods of making seals; such a method comprises the following steps: providing a rubber substrate; then applying a powdered crystalline polyolefin to the rubber substrate, in an amount sufficient to form a continuous layer when
35 melted; and then melting the powdered crystalline polyolefin to form a continuous polyolefin coating disposed on and adherent to the rubber substrate. The invention also relates to seals comprising: a polyolefin rubber substrate; and a continuous coating of fused polyolefin disposed on and adherent to the rubber substrate.

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Brief Description of the Drawings

Figure 1 shows the laminate, composed of the rubber substrate and the polyolefin coating disposed on the rubber substrate.

Figure 2 shows an embodiment of a vehicle seal composed of the rubber substrate and the polyolefin coating disposed in the glass run channel.

Figure 3 shows a another embodiment of a vehicle seal composed of the rubber substrate and the polyolefin coating disposed in the glass run channel.

10

Detailed Description of the Invention

Referring to Figure 1 the coated substrate 10 is shown, which is composed to the rubber substrate 12 and the polyolefin coating 14. Referring to Figure 2, a vehicle seal 110 is shown composed of rubber substrate, more specifically rubber body 112 and polyolefin coating 114 disposed in the glass run channel 116. Referring to Figure 3, a vehicle seal 210 is shown composed of rubber substrate, more specifically rubber body 212 having glass run channel 216. The polyolefin coating 214 is disposed on the channel face surface 218 of rubber body 212 and optionally on the glass-contacting surfaces 224 and 226 of lip 220 and lip 222 of the rubber body 212. Glass G is shown in glass run channel 216.

The coated rubber has an abrasion resistance preferably greater than 30 cycles/ μm , more preferably greater than 100 cycles/ μm , even more preferably greater than 200 cycles/ μm , most preferably greater than 300 cycles/ μm . The coating adheres well to the rubber. The coated rubber has a coefficient of friction preferably below 0.5, preferably less than 0.4, more preferably less than 0.3. Preferably the coefficient of friction is from 0.01 to 0.5, more preferably from 0.01 to 0.4, most preferably from 0.1 to 0.3.

The polyolefin coating preferably is preferably from 5 μm to 3mm, more preferably from 25 μm to 0.9 mm, in average thickness.

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Forming the Polyolefin Coating on the Rubber Substrate

The method involves coating a rubber with a crystalline polyolefin powder. The crystalline polyolefin powder is typically applied by conventional application techniques, such as, for example, by sprinkling, by dipping, by powder-dropping from a continuous feeder belt; by electrostatic spray; by running extrudate through a powder fluid bed; by drawing down by applicator, or by a powder-coating gun. Alternatively, a rubber substrate emerging from an extruder is passed through a crystalline polyolefin powder - inert gas bed. The crystalline polyolefin powder is applied to a rubber substrate, which substrate is preferably at a temperature of from -40°C to 315°C and may be applied to cured or uncured rubber substrate.

The layer of the crystalline polyolefin powder has an average thickness greater than 5µm. Once the crystalline polyolefin powder is applied to the substrate, it is heated to melt and fuse the powder. Where the crystalline polyolefin polymer has a molecular weight of greater than about 3,000,000, the crystalline polyolefin powder typically incompletely fuses upon heating, and the crystalline polyolefin powder while still molten is preferably compressed by rolling such as with a glass or metal cylinder. However, during the rolling process care is to be taken to not shift the mass of heated polyolefin coating; such shift decreases the adhesion of the polyolefin coating when the coated substrate is cooled.

Where the rubber substrate is not fresh, for example the rubber substrate displays bloom, it is preferable that the surface of the rubber substrate is cleaned before applying the crystalline polyolefin powder. The bloom is typically the result of components such as sulfur, stearic acid or anti-oxidants and the like, migrating to the surface of the rubber substrate. Conventional cleaning techniques are suitable. Good results have been obtained by abrading pre-vulcanized rubber, such as with emery paper or by solvent cleaning such as by wiping with toluene or mineral spirits or by both abrading and solvent cleaning. Where the surface is abraded, it is preferred that fine particulates produced by the abrading process be removed such as by rinsing. Good results have been obtained by using toluene. Air jets or water jets are also suitable for such rinsing.

Where vehicle sealing strips are prepared by extrusion, the polyethylene powder is preferably applied to the moving rubber extrudate after it exits the extruder die, but before the rubber

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extrudate enters a curing oven. As the rubber extrudate is heated for curing, the powder fuses and adheres to the substrate rubber. An advantage of applying the powder polyolefin to fresh rubber from the extruder is that the rubber does not require cleaning prior to the application of the powder.

Optionally, various textures, and colors are created in the polyolefin coating. Optionally, conventional pigments are added to the polyolefin coating prior to heating the coated substrate. Conventional pigments such as for example titanium dioxide, carbon black, and conventional colored pigments are suitable. Optionally, texture is imparted to the polyolefin coating by varying the powder particles size and molecular weight of the polyolefin polymer. Larger particle sizes and higher molecular weights tend to produce rougher surfaces. Optionally, texture is imparted to the surface of the coating by using a blend of differing molecular weight polyolefin polymers or different types of polyolefin polymers, such as for example a blend of LDPE and UHMWPE or LDPE and isotactic polypropylene. Optionally, texture is imparted to the surface of the coating by varying the amount of crystalline polyolefin powder initially applied to the rubber; areas with greater quantities will provide bumps whereas areas having less crystalline polyolefin powder will produce valleys. Alternatively, smooth rollers or textured rollers are impressed into the warm polyolefin coating to provide the polyolefin coating with a smooth surface or a textured surface.

25

The Rubber Substrate

The rubber substrate is flexible and comprises an aliphatic hydrocarbon polyolefin rubber. Preferably the polyolefin/aliphatic hydrocarbon rubber is unsaturated, preferably having a diene content of less than about 15 weight percent, preferably less than about 10 weight percent. Preferably, the rubber is a conventional rubber such as used in vehicle seals. Suitable rubbers are, for example, natural rubber, blends comprising a thermoplastic, crystalline polyolefin polymer and vulcanized hydrocarbon rubber particles as described in United States Patent No. 4,130,534 to Coran, et al. issued December 19, 1978; U.S. Patent No. 4,130,535 to Coran, et al. issued December 19, 1978; U.S. Patent No. 4,311,628 to Abdou-Sabet, et al. issued January 19, 1982; U.S. Patent No. 4,594,390 to Abdou-Sabet, et al. issued June 10, 1986; U.S. Patent No. 5,397,832 to Ellul issued March

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14, 1995; and U.S. Patent No. 5,290,880 to Moench, et al. issued May 1, 1994, synthetic polyisoprene rubber, polybutadiene rubber, ethylene propylene diene terpolymer (hereinafter "EPDM"), ethylene propylene rubber (hereinafter "EPR"), butyl rubber, (hereinafter "IIR"), chlorobutyl rubber, (hereinafter "CIIR") and bromobutyl rubber, (hereinafter "BIIR") The rubber polymer in the rubber substrate preferably has a weight average molecular weight of 50,000 to 2,000,000, g/mole, more preferably from 80,000 to 500,000 g/mole, most preferably from 100,000 to 300,000 g/mole.

10

The Crystalline Polyolefin Powder

The crystalline polyolefin powder comprises crystalline polyolefin polymer. The crystalline polyolefin polymer has a crystallinity X_c of preferably from 20 wt.% to 100 wt. %, more preferably from 30 wt.% to 100 wt. %, even more preferably from 40 wt.% to 100 wt. %, most preferably from 40 wt.% to 88 wt. %, as estimated from the density of the crystalline polyolefin polymer. Thus:

$$X_c = 100 \times \frac{(P_s - P_a)}{(P_c - P_a)}$$

20 where P_s is the density of the sample, P_a is the pure crystal density (1.000) and P_c is the pure amorphous density (0.85).

The crystalline polyolefin powder preferably has an average particle diameter of 600 μm or greater, and preferably 5 μm or greater. Preferably, the crystalline polyolefin powder particle size is from 5 μm to 600 μm , more preferably from 10 μm to 350 μm ; most preferably from 90 μm to 250 μm .

The crystalline polyolefin powder preferably has an maximum particle size of 1 mm or less, and preferably 600 μm or less. Preferably the maximum crystalline polyolefin powder particle size is from 10 to 1mm, more preferably from 15 μm to 600 μm , even more preferably from 20 μm to 500 μm ; most preferably from 25 μm to 300 μm .

The crystalline polyolefin powder preferably has a melt flow index at 190°C under a load of 2 kg, from 0.0 to 100 g/10 minutes, more preferably from 0.0 to 50 g/10 minutes, and even more preferably from 0.0 to 25 g/10 minutes. The crystalline polyolefin powder preferably has a melt flow index at 190°C under a load of 5 kg, from 0.0 to 200 g/10 minutes, more preferably from 0.0 to 100 g/10 minutes, and even more preferably from 0.5 to 50 g/10 minutes. The

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crystalline polyolefin powder preferably has a melt flow index at 190°C under a load of 21.6 kg, from 0.0 to 500 g/10 minutes, more preferably from 5.0 to 250 g/10 minutes, and even more preferably from 10 to 100 g/10 minutes.

- 5 The crystalline polyolefin polymer preferably has a melting point greater than 100°C but less than the decomposition point of the rubber substrate.

 The polyolefin polymer is preferably a homopolymer or copolymer of polypropylene or of polyethylene or mixtures thereof.

- 10 Where the crystalline polyolefin powder comprises polyethylene, the crystalline polyolefin powder comprises at least one high density polyolefin polymer, having a weight average molecular weight of 30,000 to 10,000,000 g/mole, more preferably from 30,000 to 1,000,000 g/mole, even more preferably 100,000 to 600,000 g/mole,
15 most preferably from 200,000 to 400,000 g/mole.

- As used herein, the term "high density polyethylene" means conventional high density polyethylene polymers as well as conventional ultra-high molecular weight linear polyethylene polymers, and thus "high density polyethylene" as used herein
20 includes linear polymers having a density of 0.94 to 0.97, as well as linear polymers having a molecular weight of 3,000,000 or higher, and having a density of 0.93 or higher.

- As used herein, the term "low density polyethylene" means conventional low density polyethylene polymers as well as
25 conventional medium density branched polyethylene polymers, and thus "low density polyethylene" as used herein includes branched polyethylene polymers having a density of 0.915 to 0.93, as well as branched polyethylene polymers having a density of 0.89 to 0.94.

- High density polyethylene polymers having molecular weights
30 above 3,000,000 tend to produce coatings which are incompletely fused and do not adhere well to the substrate. Preferably a second polyethylene polymer is therefore added to such ultra high molecular weight polyethylene polymer. Such second polyethylene polymer is either a low density polyethylene polymer having a weight average
35 molecular weight of preferably from 30,000 to 150,000 g/mole or a high density polyethylene polymer having a weight average molecular weight of preferably from 10,000 to 2,000,000 g/mole, more preferably from 30,000 to 150,000 g/mole.

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Where the polyolefin comprises polypropylene, the polypropylene is preferably isotactic and preferably has a density from 0.880 to 0.92 and a crystallinity preferably between 30% and 100% by weight.

In one preferred embodiment, the crystalline polyolefin powder comprises a blend of crystalline polyolefin powders; specifically the crystalline polyolefin powder comprises from 10 to 90 parts by weight of a powdered high-density polyethylene polymer having a weight-average molecular weight between 200,000 and 10,000,000 g/mol., and 90-10 parts by weight of a powdered high- or low-density polyethylene polymer, having a weight-average molecular weight between 30,000 and 150,000 g/mole.

In another preferred embodiment, the crystalline polyolefin powder comprises a blend of crystalline polyolefin powders; specifically the crystalline polyolefin powder comprises 25 to 75 parts by weight of a powdered high-density polyethylene polymer, having a weight-average molecular weight between 250,000 and 6,000,000 g/mol., and 75 to 25 parts by weight of powdered high density polyethylene polymer, having a weight average molecular weight between 40,000 and 150,000 g/mole.

Generally, where the crystalline polyolefin powder comprises only one crystalline polyolefin polymer, it is preferred that such polyolefin polymer is a high density polyethylene having a weight average molecular weight of from 30,000 to 3,000,000 g/mole, more preferably from 100,000 to 1,000,000 g/mole, even more preferably from 200,000 to 600,000 g/mole, most preferably from 200,000 to 400,000 g/mole.

Polyolefin polymers having a molecular weight below 30,000 g/mole tend to produce coatings displaying high wear rate, and are not preferred.

Examples of crystalline polyolefin polymers suitable alone or in a blend to form the polyolefin coating are listed in Tables IA, IB and 1C below.

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Table IA

CRYSTALLINE POLYOLEFIN POWDERS

Property	Hostalen GHR 8110 UHMWHDPE	Hostalen GHR 8020 UHMW HDPE	Hostalen GUR400F UHMW HDPE	Coathylene HA 1931 LDPE	Hostalen GUR X117 UHMW HDPE	HDPE Spectr- oscopy Grade MP*: 130- 145°C
	Ticona	Ticona	Ticona	Composite Particles	Ticona	Aldrich
MFI A	-		-	2	-	-
MFI B	-	0.5	-	-	-	-
MFI C	0.8-1.6	14	<1		<1	-
Density (g/cm ³)	0.95	0.95	0.93	0.919	0.93	-
Crystall- ine melting range (°C)	-	130-135	130-135	108-113	130-135	130-140
Vicat softenin g point (°C)	-	-	-	87	-	-
Number avg. mw (g/mole)		-	-	32,000	-	-
Weight avg. mw (g/mole)	600,000	300,000	more than 6,000,00 0	730,000	4,400,000	-
Max. particle size (d) micro- meters	<500	-	125	75	125	-
Mid- range particle size	120	210	c. 60	17	c. 60	-
Mesh size	-	-	-	200	-	-

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Table IB

CRYSTALLINE POLYOLEFIN POWDERS

Property	UHMW PE	UHMW PE Treated Surface	Coathyl- ene NB6454 HDPE	Coathyl- ene NC5374F HDPE	Coathyl- ene HA2454 LDPE*	Coathyl- ene HO1681 LDPE
	Aldrich	Aldrich	Clariant	Clariant	Clariant	Clariant
MFI A	-	-	8	20	7	70
MFI B	-	-	-	-	-	-
MFI C	-	-	-	-	-	-
Density (g/cm ³)	0.94	0.94	0.964	0.953	0.9244	0.916
Crystall- ine melting range (°C)	138	138	128-134	126-133	108-113	102-109
Vicat softening point (°C)	-	-	127	124	91	70
Number avg. mw (g/mole)	-	-	25,000	18,000	30,000	21,000
Weight avg. mw (g/mole)	3,000,00 0- 6,000,00 0	-	80,000	60,000	67,000	295,000
Max. particle size (d) micro- meters	-	-	90	125	75	630
Mid-range particle size	-	180	30	75	17	305
Mesh size	-	-	170	120	200	38

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Table IC
CRYSTALLINE POLYOLEFIN POWDERS

Property	MDPE* MP*: 109-111	MICRO- SCRUB 50 HMW* LDPE	Coathyle ne PY0787F homo-PP*	Coathyle ne PB0580 homo-PP	Propyl- matte 31 powder PP
	Aldrich	Micro Powders	Clariant	Clariant	Micro Powders
MFI A.	-	-	-	-	-
MFI B.	-	-	60	100	-
MFI C.	-	-	-	-	-
Density (g/cm ³)	0.94	-	0.907	0.905	0.86
Crystallin e melting range (°C)	109-111	107-109	162-168	162-168	160-170
Vicat softening point (°C)	-	-	148	145	-
Number avg. mw (g/mole)	-	-	-	-	-
Weight avg. mw (g/mole)	-	-	-	-	-
Max. particle size (d) micro- meters	-	297	200	90	31
Mid-range particle size micro- meters	-	-	110	38	12
Mesh size	-	50	70	170	

*MP - melting point melting range

5 MFI A - melt flow index (MI 190°C/2kg)g/10 minutes

MFI B - melt flow index (MI 190°C/5kg)g/10 minutes

MFI C - melt flow index (MI 190°C/21.6kg)g/10 minutes

Manufacturer's designations:

10 HMW = high molecular weight,

UHMW = ultra-high molecular weight,

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HDPE = high-density polyethylene,
MDPE = medium-density polyethylene
PE = polyethylene,
LDPE = low-density polyethylene
5 PP = isotactic polypropylene

The polyethylene polymer available as Vistomer HD 2800 from Composite Particles Inc. is coated with a proprietary adhesive by the manufacturer and has a melt flow index at 190°C under a load of 21.6 10 kg, of less than 1 g/10 minutes, a density of 0.93 g/cm³, a crystalline melting range of 130-135, a weight average molecular weight of more than 6,000,000 a maximum particle size of 125 microns, a mid range particle size of c.60 and a mesh size of 8,000. The Vistomer HD 2800 polyethylene is less preferred particularly for use 15 in a vehicle seal.

The following examples illustrate the invention and are not intended to be limiting.

The crystalline polyolefin powder was typically applied by one of two methods; by dipping or by drawing the crystalline polyolefin 20 powder along the substrate with a tool. The dipping method involved placing the rubber substrate into the crystalline polyolefin powder to obtain about 0.2 to 2 mm layer of the powder on the substrate. The crystalline polyolefin powder was applied by dipping in Examples 3, 4, 6, 7, 18, 20-25, 33, 40 and 41.

25 Alternatively, about 1 gram of crystalline polyolefin powder was placed near one end of a 20 cm x 2 cm rubber strip. A notched tool was used to draw down a strip of powder down the length of the strip. The tool, made from a sheet of a high-hardness rubber had a notch 1 cm wide and 2 mm deep. The tool applied a strip of 30 crystalline polyolefin powder 2 cm wide and 1 to 2 mm thick and about 1 gram in weight, to the rubber substrate.

Example 1

A coated substrate was prepared as follows. The surface of a 35 piece of vulcanized, carbon black filled EPDM rubber was cleaning/roughening by abrading the surface by stroking the surface sheet about 20 times with emery paper. Then the surface was scrubbed with toluene for a short time and wiped dry. After a few minutes when the surface appeared to be dry by visual examination, the powder 40 was applied.

The substrate was heated for 5 minutes at 230°C in an air circulating oven. Then a layer of polyethylene powder sold under the

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trade name Hostalen GHR 8020 from Ticona, was applied by dipping the rubber substrate into the powder to form a layer of powder approximately 1mm thick at the thickest points. The substrate was then placed in a 230°C oven for 5 minutes and removed and cooled.

5 Example 2

A coated substrate was prepared as in Example 1, except that after the coated substrate was removed from the oven, a room temperature glass cylinder or a steel cylinder was pressed and rolled over the molten polyolefin, by hand, for about 15 seconds. The
10 cylinder was then removed; the resulting coated rubber was smooth and even.

Example 3

A coated substrate was prepared as in Example 2, except that about one half the amount of crystalline polyolefin powder was used.

15 Example 4

A coated substrate was prepared as in Example 3.

Example 5

A coated substrate was prepared as in Example 2.

Example 6

20 A coated substrate was prepared as in Example 1, except that the Polymatte 31 polypropylene was used instead of the GHR8020.

Example 7

A coated substrate was prepared as in Example 6.

Example 8

25 A coated substrate was prepared as in Example 2, except that the GURX117 polyethylene was used.

Example 9

A coated substrate was prepared as in Example 1, except that Microscrub 50 polyethylene was used.

30 Example 10

A coated substrate was prepared as in Example 1.

Example 11

A coated substrate was prepared as in Example 2, except that Microscrub 50 polyethylene was used.

35 Example 12

A coated substrate was prepared as in Example 2, except that a mixture of 60% by weight of the Microscrub 50 polyethylene and 40% by weight GHR 8020 polyethylene was used.

Example 13

40 A coated substrate was prepared as in Example 1, except that

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GHR 8020 polyethylene was applied to an uncured substrate and thus not pre-cleaned.

Example 14

A coated substrate was prepared as in Example 2, except that the GUR 400F polyethylene applied to an uncured substrate and thus not pre-cleaned.

Example 15

A coated substrate was prepared as in Example 2, except that GURX117 polyethylene was used.

10 Example 16

A coated substrate was prepared as in Example 2.

Example 17

A coated substrate was prepared as in Example 2, except that GHR 8110 polyethylene was used.

15 Example 18

A coated substrate was prepared as in Example 2, except that a mixture of 60% by weight of the 8020 polyethylene and 40% by weight MS50 polyethylene was used.

Example 19

20 A coated substrate was prepared as in Example 2, except that a mixture of 50% by weight of the 8020 polyethylene and 50% by weight MS50 polyethylene was used.

Example 20

A coated substrate was prepared as in Example 1, except that a mixture of 50% by weight of the 8020 polyethylene and 50% by weight MS50 polyethylene was used.

Example 21

A coated substrate was prepared as in Example 1, except that a mixture of 25% by weight of the 8020 polyethylene and 75% by weight MS50 polyethylene was used.

Example 22

A coated substrate was prepared as in Example 22.

Example 23

A coated substrate was prepared as in Example 1, except that a thin layer of an ultra high molecular weight polyethylene from Aldrich was used.

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Example 24

A coated substrate was prepared as in Example 1, except that "high density polyethylene" spectroscopy grade 130-145°C from Aldrich was used.

5 Example 25

A coated substrate was prepared as in Example 1, except that a "medium density polyethylene" melting point 109-111°C from Aldrich was used.

Example 26

10 A coated substrate was prepared as in Example 2, except that ultra high molecular weight polyethylene from Aldrich was used.

Example 27

A coated substrate was prepared as in Example 2, except that ultra high molecular weight polyethylene, having a treated surface, 15 from Aldrich was used.

Example 28

A coated substrate was prepared as in Example 2, except Vistamer HD 2800 polyethylene coated with a manufacturer's proprietary adhesive, was used.

20 Example 29

A coated substrate was prepared as in Example 1, except Vistamer HD 2800 polyethylene, coated with a manufacturer's proprietary adhesive, was used.

Example 30

25 A coated substrate was prepared as in Example 2, except that a the NC5374 Coathylene polyethylene was used.

Example 31

A coated substrate was prepared as in Example 2, except that a the Coathylene NB6454 polyethylene was used.

30 Example 32

A coated substrate was prepared as in Example 2, except Coathylene H01681 polyethylene with MFI of 70 was used.

Example 33

A coated substrate was prepared as in Example 2, except that 35 Coathylene HA2454 polyethylene was used.

Example 34

A coated substrate was prepared as in Example 2, except that Coathylene HA1931 polyethylene from Clariant was used.

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Example 35

A coated substrate was prepared as in Example 2, except that Coathylene PB0580 polypropylene from Clariant was used.

Example 36

5 A coated substrate was prepared as in Example 2, except that Coathylene PB0580 polypropylene from Clariant was used.

Example 37

A coated substrate was prepared as in Example 2, except that Coathylene PY0787 polypropylene from Clariant was used.

10 Example 38

A coated substrate was prepared as in Example 2, except that a mixture of 100 parts by weight of the 8020 polyethylene and 6.5 parts by weight molybdenum disulfide powder was used.

Example 39

15 A coated substrate was prepared as in Example 1, except that a mixture of 100 parts of the MS50 polyethylene and 6.5 parts by weight molybdenum disulfide powder was used.

Example 40

A coated substrate was prepared as in Example 2, except that a mixture of 50% by weight of the MS50 polyethylene and 50% by weight GHR 8020 polyethylene along with 6 parts molybdenum disulfide powder was used.

Example 41

25 A coated substrate was prepared as in Example 2, except that a mixture of 25% by weight of the N6454 polyethylene and 75% by weight of the 8110 polyethylene was used.

Example 42

A coated substrate was prepared as in Example 2, except that a mixture of 25% by weight of the N5374 polyethylene and 75% by weight GHR 8110 polyethylene was used.

30 Example 43

A coated substrate was prepared as in Example 2, except that a mixture of 50% by weight of the N5374 polyethylene and 50% by weight GHR 8110 polyethylene was used.

35 Example 44

A coated substrate was prepared as in Example 1, except that a mixture of 50% by weight of the N6454 polyethylene and 50% by weight of the 8110 polyethylene was used.

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Example 45

A coated substrate was prepared as in Example 1, except that a mixture of 75% by weight of the N5374 polyethylene and 25% by weight GHR 8020 polyethylene was used.

5 Example 46

A coated substrate was prepared as in Example 1, except that the GHR 8110 polyethylene was used.

Example 47

10 A coated substrate was prepared as in Example 1, except that a mixture of 25% by weight of the N5374 polyethylene and 75% by weight GHR 8110 polyethylene was used.

Example 48

15 A coated substrate was prepared as in Example 1, except that a mixture of 38% by weight of the N5374 polyethylene and 62% by weight GHR 8110 polyethylene was used.

Example 49

A coated substrate was prepared as in Example 1, except that N5374 polyethylene was used.

Example 50

20 A coated substrate was prepared as in Example 1, except that a mixture of 75% by weight of the N5374 polyethylene and 25% by weight GHR 8100 polyethylene was used.

Example 51

25 A coated substrate was prepared as in Example 1, except that a mixture of 50% by weight of the N5374 polyethylene and 50% by weight GHR 8100 polyethylene was used.

Comparative Example A An EPDM rubber strip without a coating was used as a control.

30 Comparative Example B An EPDM rubber strip with a Versicoat® polyurethane as coating was prepared using conventional methods.

Comparative Example C A coated substrate was prepared as in Example 1, except that a thin layer less than 300µm of GHR 8020 polyethylene was used.

35

Evaluation

The coated rubbers of the Examples and the Comparative Example were evaluated for abrasion resistance, coefficient of friction and adhesion. After cooling, the test strips were tested by using the
40 crockmeter. The coefficient of friction was recorded as a function

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of the number of crockmeter cycles. The typical average thickness of the films prepared was about 20 mils (about 0.5 mm).

The durability of the samples was determined by test method entitled General Motors Engineering Standards, Seals Abrasion Resistance GM9909P:R1:ETSSLN, January 1993. The abrasion resistance was measured by using the wear resistance testing apparatus, as shown in Figure 1 of the General Motors Standards. A 4 mm thick piece of non-tempered glass having a radius on the edge of 2.5 to 6 mm, with satin finish edge was rubbed, under weight, back and forth across the coated substrate. The samples of the coated substrates of the examples, were cut to 200 mm in length and trimmed as needed. The samples were mounted to a mounting fixture which was then attached to the wear resistance testing apparatus. The mounting fixture was centered and tighten so that the mounting fixture remained stationary and straight during the test. A weight of 2.7 kilograms was applied and the wear resistance testing apparatus was set to 60 cycles per minute, where the stroke of the abrasion element, that is, the glass, is 150 mm with one back and forth movement as one cycle. The glass was loaded to the wear resistance tester as per profile print in Figure 1 of the General Motors Engineering Standards.

The wear resistance test was performed and the samples were typically visually examined every 500 cycles. The glass was replaced every 5000 cycles.

In addition to visual examination, the depth of wear was determined by optical microscopy. Thus, the wear resistance is reported as number of cycles per micrometer (micron). The results are presented in Table II.

The coefficient of friction was determined by dividing lateral force to move the glass by the normal force 2.7 kg. this was done for each cycle and averages were taken. The polyolefin coatings of the Examples were evaluated for adhesion to the rubber substrate by visual examination and manual examination.

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Table II

SUMMARY OF CROCKMETER AND WEAR RESULTS

Sample	Powder Type	Initial COF	Avg. COF	Cycles To fail	Coating Appearance	Depth of Wear, micrometers	Abrasion Resistance cycles/micrometer
Comparative Ex. _	Control	0.7027	0.6775	8640	None	--	--
Comparative Ex. _	Control	0.6584	0.6204 1	5000	None	--	--
Comparative Ex. _	HS-37-EX-2 (Versicoat PU)	0.3093	0.4262	1314	--	--	--
Ex. 1	Hostalen GHR8020 not rolled	0.277	0.3461	16600	Moderately heavy uniform, rough	WT	--
Ex. 2	Hostalen GHR8020 rolled	0.3208	0.4351	3300	Medium thick, uniform, rough	WT	--
Ex. 3	Hostalen GHR8020 rolled	0.4038	0.4497	430	Light, uniform, rough	52	8.3
Ex. 4	Hostalen GHR8020	0.3714	0.3946	4835	Light to medium, slightly rough	75	64.5
Ex. 5	Hostalen GHR8020 rolled	0.3171	0.3455	5000	Medium to heavy, slightly rough	59	84.7
Ex. 6	Polymatte 31	0.7354	0.7353	106	Moderate to thin, smooth	WT	--
Ex. 7	Polymatte 31	0.7758	0.7758	115	Moderate to thin, smooth	WT	--
Ex. 8	Hostalen GURX117	0.3168	0.4149	209	Medium thick uniform unfused	WT	--
Ex. 9	Microscrub 50 not rolled	0.4663	0.515	244	Aesthetically mottled	WT	--
Ex. 10	Hostalen GHR8020 not rolled	0.2475	0.2823	20000	Thick, smooth, translucent	153	130.7

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Sample	Powder Type	Initial COF	Avg. COF	Cycles To fail	Coating Appearance	Depth of Wear, micro-meters	Abrasion Resistance cycles/micrometer
Ex. 11	Microscrub 50 rolled	0.4404	0.464	10000	Thick, smooth, translucent	WT	--
Ex. 12	Microscrub 50/Hostalen GHR8020 60/40 rolled	0.3657	0.3409	20000	Thick, smooth, translucent	257	77.8
Ex. 13	Hostalen GHR8020#	0.295	0.3061	20000	Thick, smooth, translucent	62	322.6
Ex. 14	Hostalen GUR400# rolled	0.2402	0.3186	20000	Smooth, white film, poor adhesion	33	606.1
Ex. 15	Hostalen GURX117 rolled	0.3322	0.3889	6675	Smooth, white film; poor adhesion	20	333.8
Ex. 16	Hostalen GHR8020 rolled	0.3593	0.3229	20000	Thick, smooth, translucent	129	155.0
Ex. 17	Hostalen GHR8110 rolled	0.2371	0.255	20000	Thick, translucent	31	645.2
Ex. 18	GHR8020/Microscrub 50 60/40 rolled	.05321	0.34	10000	White, moderately thick	43	232.6
Ex. 19	Hostalen GHR8020/Microscrub 50 50/50 rolled	0.4698	0.4563	4153	Moderately thin, translucent	96	43.3
Ex. 20	Hostalen GHR8020/Microscrub 50 25/75 not rolled	0.3814	0.4364	2334	Medium thin rough, translucent	WT	--
Ex. 21	Hostalen GHR8020/Microscrub 50 25/75 not rolled	0.3676	0.5592	1000	Medium thin rough, translucent	WT	--

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Sample	Powder Type	Initial COF	Avg. COF	Cycles To fail	Coating Appearance	Depth of Wear, micrometers	Abrasion Resistance cycles/micrometer
Ex. 22	Hostalen GHR8020/ Microscrub 50 25/75 not rolled	0.4782	0.4928	6146	Medium thin rough, translucent	WT	--
Ex. 23	UHMW PE (Aldrich)	1.1185	1.0854	59	Very thin, milky white	WT	--
Ex. 24	HDPE Spectroscopy Grade MP: 130-145 °C, (Aldrich)	0.4545	0.5719	246	Thin, smooth transparent	WT	--
Ex. 25	Medium density PE (MDPE) MP: 109-111, (Aldrich)	0.5158	0.5588	2487	Medium thickness, transparent bumpy	WT	--
Ex. 26	PE UHMW (Aldrich) rolled	0.4945	0.5439	121	Thick white film, poor adhesion	32	3.8
Ex. 27	PE UHMW (Aldrich), treated surface not rolled	0.3007	0.3508	10000	Thick off-white film, poor adhesion	34	294.1
Comparative Ex. 28	Vistomer HD2800 rolled	0.3976	0.4096	85	Smooth, thick translucent film	40	2.1
Ex. 29	Vistomer HD2800, rolled	0.3667	0.381	5670	Smooth, thick, transparent film	WT	--
Ex. 30	Coathylene NC5374 rolled	0.22	0.2152	10000	Smooth, transparent, cracks in groove after test	97	103.1
Ex. 31	Coathylene NB6454 rolled	0.2077	0.2134	10000	Smooth, transparent puddles	126	79.4
Ex. 32	Coathylene HO1681 rolled	0.4499	0.5867	6390	Smooth, transparent, thin	WT	--

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Sample	Powder Type	Initial COF	Avg. COF	Cycles To fail	Coating Appearance	Depth of Wear, micrometers	Abrasion Resistance cycles/micrometer
Ex. 33	Coathylene HA2454 rolled	0.4486	0.4526	62	Smooth, transparent, puddles	68	0.9
Ex. 34	Coathylene HA1931 rolled	0.3389	0.4275	1240	Smooth, transparent, puddles	348	3.6
Ex. 35	Coathylene PB0580 rolled	0.3545	0.4016	4259	Fairly thick, uniform, rough	140	30.4
Ex. 36	Coathylene PB0580	0.4386	0.4778	180	Smooth, transparent	113	1.6
Ex. 37	Coathylene PY0787 rolled	0.2046	0.3029	3108	Smooth, thick, transparent film	78	39.8
Ex. 38	Hostalen GHR8020/ MoS ₂ 100/6.5 rolled	0.296	0.3298	10000	Medium thickness, uniform, rough	87	114.9
Ex. 39	Microscrub 50/ MoS ₂ 100/6.5	0.5217	0.5225	63	Thin, mottled	59	1.1
Ex. 40	Microscrub 50/ Hostalen GHR8020/ MoS ₂ rolled	0.3884	0.3818	10000	Moderately thin, translucent	121	82.6
Ex. 41	NB6454/ GHR8110 25/75 rolled	0.2061	0.2351	10000	Thick, uniform, rough	42	238.1
Ex. 42	Coathylene NC5374/ GHR8110 25/75 rolled	0.2438	0.2512	10000	Thick, uniform, rough	66	151.5
Ex. 43	Coathylene NC5374/ GHR8110 50/50 rolled	0.2538	0.2613	10000	Thick, uniform, rough	89	112.4

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Sample	Powder Type	Initial COF	Avg. COF	Cycles To fail	Coating Appearance	Depth of Wear, micrometers	Abrasion Resistance cycles/micrometer
Ex. 44	Coathylene NB6454/ Hostalen GHR8110 50/50 rolled	0.1945	0.217	10000	Thick, uniform, rough	40	250.0
Ex. 45	Coathylene NC5374/ GHR8110 75/25 not rolled	0.2515	0.2816	10000	Thick, uniform, rough	83	120.5
Ex. 46	Hostalen GHR8110 not rolled	0.2724	0.2575	10000	Thick, uniform, rough	115	87.0
Ex. 47	Coathylene NC5374/ GHR8110 25/75 not rolled	0.2227	0.2165	10000	Thick, uniform, rough	45	222.2
Ex. 48	Coathylene NC5374/ GHR8110 38/62 not rolled	0.2136	0.246	10000	Thick, uniform, rough	76	131.6
Ex. 49	Coathylene NC5374 not rolled	0.3462	0.3431	5100	Bumpy	WT	--
Ex. 50	Coathylene NC5374/ GHR8110 75/25 not rolled	0.2782	0.2847	10000	Thick, uniform, rough	209	47.8
Ex. 51	Coathylene NC5374/ GHR8110 50/50 not rolled	0.2136	0.2182	10000	Thick, uniform, rough	73	137.0

WT - worn through

rubber substrate not cured at time crystalline polyolefin powder was applied

5 The coatings of the Examples show reduced coefficients of friction in comparison to the comparative Example. In particular, the coatings of Examples 9, 10, 11, 13, 14b, 14/18, 15-17, 19 21, 22, 23b, have significantly reduced coefficients of friction and show good wear characteristics. When the Versicoat® polyurethane control 10 coating is fresh, the wear resistance is typically about 400 cycles/micron. However, with weathering, the wear resistance

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typically drops to about 10 cycles/micron, and the coefficient of friction greatly increases.

Thus, a reduced coefficient of friction is obtained with the coated substrates of the present invention. The wear resistance of the coating containing a polyethylene polymer having a molecular weight of 300,000 and higher, Examples 13 to 17 is greater than coatings containing polyethylene with a molecular weight below 300,000, such as Examples 30 and 31. However, the adhesion of the ultra high molecular weight HDPE to the substrate is typically not as strong as with the other HDPE.

Surprisingly, the coating which is a mixture of the of ultra-high molecular weight HDPE and the lower-molecular-weight HDPE has improved wear resistance and satisfactory adhesion to the substrate.

The coated substrate prepared in example 14, employed a polyethylene polymer having a molecular weight in excess of 6,000,000, GUR 400F. The resulting laminate had poor adherence making it less preferred for a vehicle seal but useful for a laminate having a temporary or easily removable polyolefin coating.

Crystalline polyolefin powders were also applied to rubber substrates having a temperature in the range of 20°C to 260°C; the coatings were satisfactory based on visual evaluation.

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What is claimed is:

1. A method of coating polyolefin rubber substrate comprising the following steps:
 - 5 a. providing a crystalline polyolefin powder comprising a crystalline polyolefin polymer having weight average molecular weight of from 30,000 to 10,000,000 and a degree of crystallinity of 20 wt.% to 100 wt.%;
 - 10 b. applying a crystalline polyolefin powder, to the polyolefin rubber substrate, in an amount sufficient to form a continuous layer when melted; and
 - c. then melting the crystalline polyolefin powder to form a continuous, fused, polyolefin coating disposed on, and adherent to, the polyolefin rubber
15 substrate.
2. The method of 1, further comprising the step of:
compressing the molten polyolefin coating.
- 20 3. The method of 1, wherein the polyolefin polymer has a an average particle diameter of from 5 to 600 μm and a melt flow index above from 0.0 to 500 g/10 minutes, at 190°C under a load of 21.6 kg., and the rubber substrate is selected from the group consisting of: ethylene propylene diene terpolymer,
25 ethylene propylene rubber, butyl rubber, chlorobutyl rubber, and bromobutyl rubber, and mixtures thereof.
4. The method of claim 3, wherein the crystalline polyolefin powder comprises a powdered polyethylene comprising at least
30 one high density polyethylene polymer.
5. The method of claim 3, wherein the crystalline polyolefin powder comprises a powdered polypropylene polymer.
- 35 6. The method of 3, wherein, wherein the crystalline polyolefin polymer comprises isotactic polypropylene.
7. The method of claim 3, wherein the crystalline polyolefin powder comprises:
40 from 10 to 90 parts by weight of powdered high-density

-25-

polyethylene polymer, having a weight-average molecular weight from 300,000 to 10,000,000 g/mol.; and

from 10 to 90 parts by weight of powdered high-density polyethylene polymer, having a weight-average molecular weight from 40,000 to 150,000 g/mol.

8. The method of claim 6, wherein the rubber substrate is ethylene propylene diene terpolymer.

9. The method of claim 3, wherein the crystalline polyolefin powder comprises:

from 25 to 75 parts by weight, of the powdered high-density polyethylene polymer, having a weight-average molecular weight from 600,000 to 6,000,000 g/mol.; and

from 25 to 75 parts by weight of powdered high-density polyethylene polymer, having a weight-average molecular weight from 40,000 to 150,000 g/mole.

10. The method of claim 4, wherein the average particle diameter of the crystalline polyolefin powder is from 30 to 350 μm .

11. The method of 4, wherein the polyolefin polymer has a weight average molecular weight of from 30,000 to 6,000,000.

12. The method of claim 9, wherein the rubber substrate is ethylene propylene diene terpolymer.

13. The method of claim 3, wherein the rubber substrate has a glass run channel therethrough and the crystalline polyolefin powder is disposed in the glass run channel and the polyolefin coating has an average coefficient of friction against glass of less than 0.5 and a wear resistance of at least 30 cycles per micrometer as measured using glass having with satin edge, under a 2.7 kg load.

35

14. The method of claim 13, wherein the polyolefin coating has an average coefficient of friction against glass of less than 0.4 and a wear resistance of at least 100 cycles/ μm as measured by using glass having with satin edge, under a 2.7 kg load.

40 load.

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15. The method of claim 14, wherein the average particle diameter of the powdered polyolefin is from 90 to 250 μm .

5 16. The method of claim 14, wherein the polyolefin coating has a wear resistance of at least 200 cycles/ μm as measured by using glass having with satin edge, under a 2.7 kg load.

17. The method of claim 14, wherein the polyolefin coating
10 has a wear resistance of at least 300 cycles/ μm as measured by using glass having with satin edge, under a 2.7 kg load.

18. The seal of claim 14, wherein the polyolefin coating has an average thickness of 10 μm to 3mm.

15

19. A vehicle seal comprising:

an rubber body having a glass run channel
therethrough, said channel defined by glass- contacting
surfaces of the rubber body; and

20

a continuous coating of fused polyolefin disposed
on at least a portion of the glass contacting surfaces
and adherent to the polyolefin rubber body; said coating
comprising a crystalline polyolefin polymer having weight
average molecular weight of from 30,000 to 10,000,000
25 and a degree of crystallinity of 20 wt.% to 100 wt.%.

25

20. A laminate comprising:

an polyolefin rubber substrate; and

a continuous coating of fused polyolefin disposed
on and adherent to the polyolefin rubber substrate said
30 coating comprising a crystalline polyolefin polymer
having weight average molecular weight of from 30,000 to
10,000,000 and a degree of crystallinity of 20 wt.% to
100 wt.%.

30

1/2

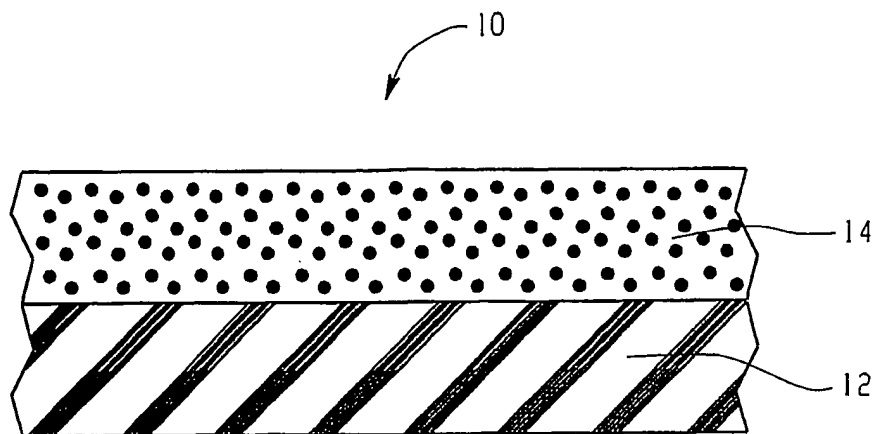


Fig. 1

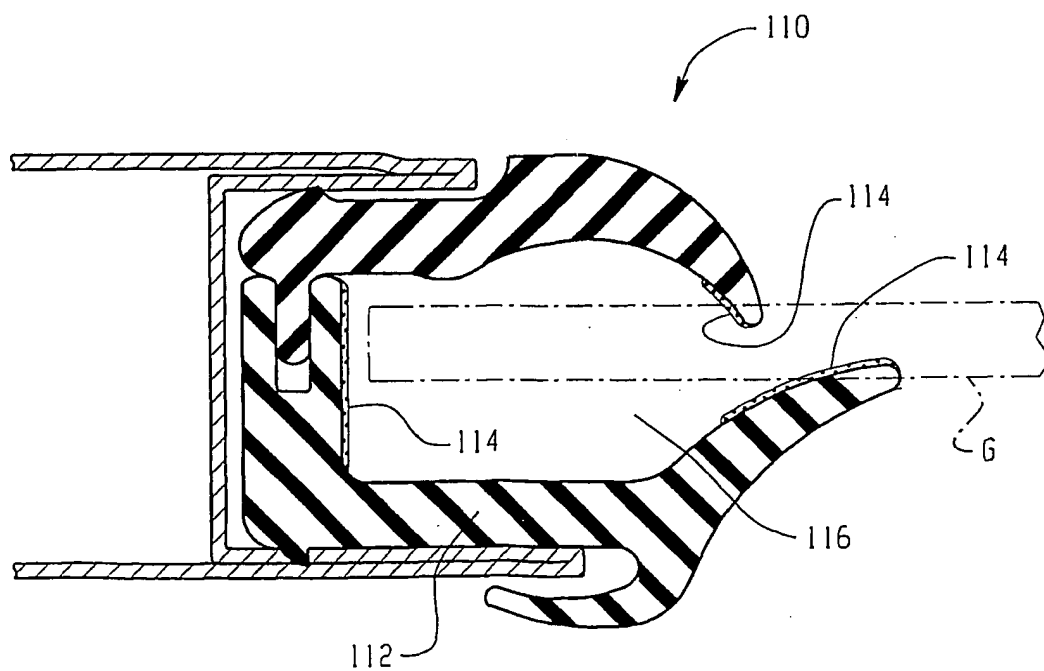


Fig. 2

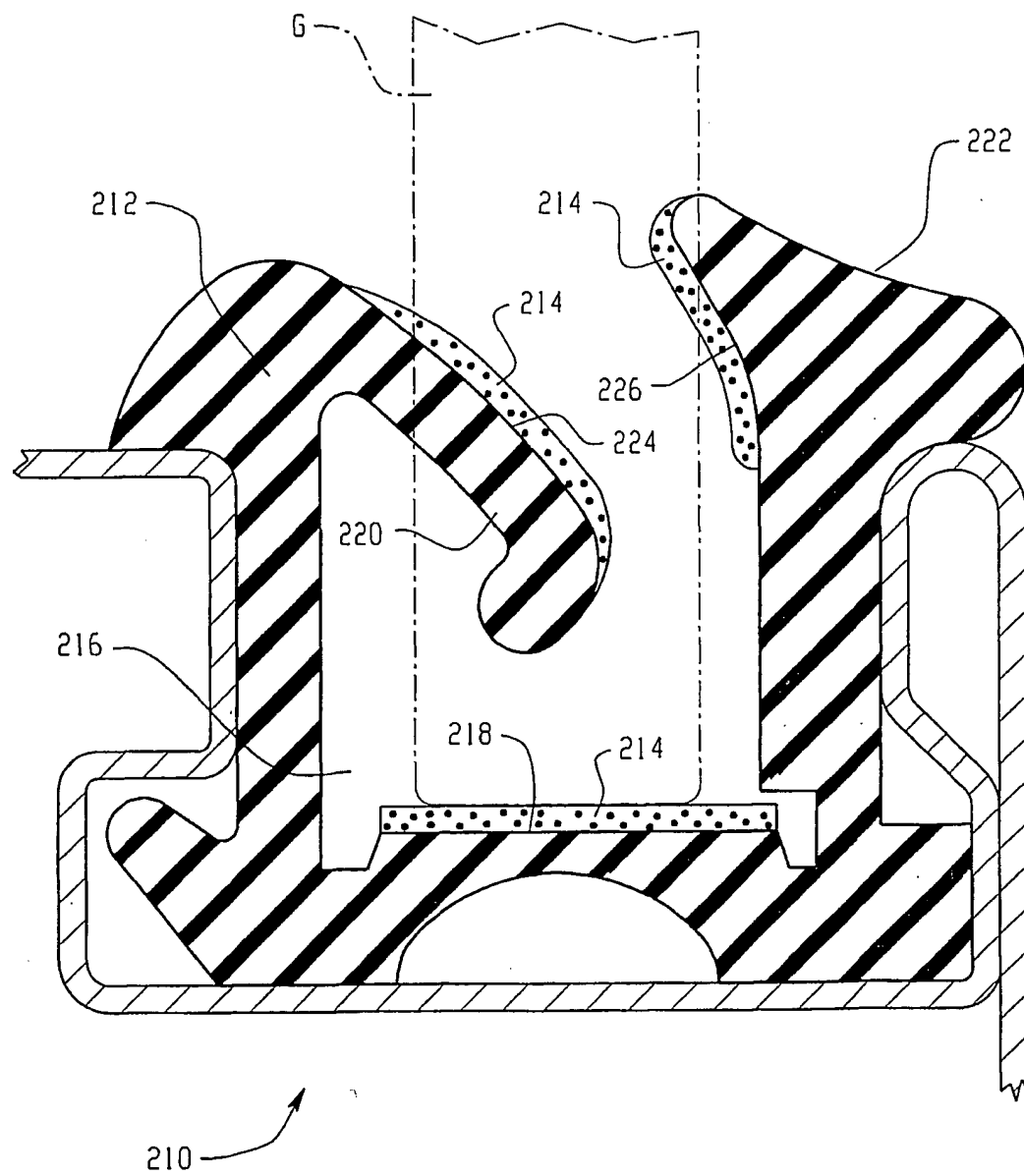


Fig. 3

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US01/14621

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : B60J 10/04; B32B 27/08, 27/32; E05D 3/02.

US CL : 428/122, 188, 192, 516; 49/441, 440, 489.1.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 428/122, 188, 192, 516; 49/441, 440, 489.1.

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,306,537 A (GUSTAFSON et al) 26 April 1994, col. 1, lines 55-61, col. 2, lines 58-66, col. 3, lines 27-40.	1-20
Y	JP 58155918 A (ASAHI YUKIZAI KOGYO KK) 16 September 1983, Abstract.	2
Y	US 4,035,528 A (POPPE et al) 12 July 1977, col. 2, lines 22-28.	1-20
Y	US 4,224,379 A (ICHINOSE et al) 23 September 1980, col. 6, lines 27-34.	4, 6
Y, P	US 6,146,739 A (ITOH et al) 14 November 2000, col. 3, lines 24-47.	7, 9
Y	US 3,891,499 A (KATO et al) 24 June 1975, col. 7, lines 61-68, col. 8, lines 1-23.	1-20

☒ Further documents are listed in the continuation of Box C.
 ☐ See patent family annex.

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 3,112,300 A (NATTA et al) 26 November 1963, col. 2, lines 8-20, col. 4, lines 53-75, col. 5, lines 1-29.	1-20

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